

## \* NOTICES \*

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention is a refining method of the copper sulfate which removes nickel from copper sulfate containing nickel efficiently, and obtains high grade copper sulfate.

[0002]

[Description of the Prior Art]In copper electrorefining, usually it increases rather than the copper quantity in which the direction of the copper eluted from an anode carries out electrocrystallization with a cathode about 1%, and it is necessary to remove the copper accumulated in the electrolysis solution. Although there is also the method of removing as a removing method of such surplus copper by electrolytic winning which uses an insoluble anode, there are demerits, like the quality of the electrolytic copper which electric power cost requires about 10 times compared with the usual electrolysis, and also is obtained is inferior.

[0003]An electrolysis solution is condensed and cooled and other removing methods also include the method of separating the copper sulfate which deposits by supersaturation as a crystal of five monohydrates. In this case, the copper sulfate obtained has a use which can count also upon sale, such as using it for agriculture as the liquid component and the object for sterilization of plating liquid, and can be called advantageous removing method as a removing method of surplus copper.

[0004]However, when selling a copper sulfate crystal to outside companies, the impurity contained poses a problem. Most impurities have adhered to the crystal of copper sulfate, and by carrying out pH adjustment in the stage before concentration, or washing the surface with pure water etc. after a crystal, it can remove easily and does not become a problem. However, since it is an impurity in which it conflicts in an impurity and reason nickel is most kept at arm's length to plating of an electronic industry material, in order to use it for these uses, it is necessary to lose nickel dignity even to

about 1/100 grade as compared with about 0.001 or less % of the weight and usual copper sulfate, for example. However, the effective method was not able to say the method of decreasing the amount of nickel with concentration and a crystal, since the action of nickel is almost the same as that of copper and the amount of [ which carries out an eutectoid to copper in addition to adhesion in a crystal surface ] nickel is also large.

[0005]Therefore, conventionally, in order to obtain high grade copper sulfate with little nickel, it was required for there to be much further 2 to 3 times or more, and to repeat the process of concentration and a crystal from usual copper sulfate, and it was remarkably disadvantageous in respect of the manufacturing cost, the capacity utilization rate, the man day, the product yield, etc.

[0006]

[Problem(s) to be Solved by the Invention]This invention removes economically adhesion thru/or the nickel which carried out the eutectoid to copper sulfate as mentioned above, and the copper sulfate which obtains copper sulfate of a high grade makes refining method offer.

[0007]

[Means for Solving the Problem]In order to solve the above-mentioned technical problem, in this invention, copper sulfate which contains nickel as an impurity is dissolved as solution, A nickel content used a refining method of copper sulfate which is 0.001 or less % of the weight by collecting copper sulfate which carries out separation sedimentation, when said solution is heated at not less than 80 \*\*, and making said copper sulfate make remelt and condense and recrystallize further. In this method, the amount of [ under atmospheric pressure ] boiling point becomes a maximum substantially [ as for the pH of a copper sulfate aqueous solution before heating, it is preferred that it is / or more 1.7 / 4.5 or less, and / cooking temperature ].

[0008]

[Embodiment of the Invention]In order to remove conventionally the nickel contained as an impurity in copper sulfate, the effective method was found only by repeating remelting as mentioned above, but it had become a neck in the production process.

[0009]On the other hand, this invention persons found out that precipitate arose by [ in which air bubbles generate the solution of copper sulfate from liquid under atmospheric pressure ] continuing heating 80 \*\* at a not less than 85 \*\* elevated temperature preferably. The maximum of cooking temperature is the boiling point under atmospheric pressure, and is about 100-110 \*\* substantially. A solution condenses this precipitate, it is considered to be a thing with heterogeneous copper sulfate which becomes supersaturation and deposits, after said precipitate deposits, heating is stopped, and even if it cools to a room temperature, the crystal of copper sulfate by supersaturation is not produced.

[0010]When said precipitate was analyzed, distributing about 1% of the solutions before nickel heating also found out. That is, high grade copper sulfate with little nickel can be obtained by collecting these precipitate, dissolving again and depositing the crystal of copper sulfate, without needing a reagent special in addition to heating etc.

[0011]if it will not generate whether this crystal is remelted if the pH of the copper sulfate solution before heating becomes lower in general than 1.7, and pH exceeds 4.5 in general, copper will precipitate as copper hydroxide, and precipitate of nickel hydroxide will also be generated, and the refining effects will be lost -- the thing header was carried out.

[0012]If it adjusts so that sulfuric acid etc. may be \*\*\*\*(ed) and it may become lower than 1.7 about pH even if precipitate generates, precipitate will remelt. Therefore, the pH of the solution before heating is preferred when or more 1.7 the thing to do to 4.5 or less carries out this invention.

[0013]

[Example](EXAMPLE) Pure water was added to 1.3 kg of copper sulfate crystals, and the bottom carried out thing preparation and it was made 2 l. with the solution A. pH of A was 2.0. It heated strong-stirring so that air may involve in A, after the dissolution was thoroughly maintained at not less than 85 °C for 1 hour, and liquid was evaporated to 1.9 l. Even if it cooled as it is by the concentration at this time, although not supersaturation but the crystal should not deposit, in order that the yellow-green precipitate B might generate a solubility top, it filtered this, and separated and analyzed it.

[0014]It condensed to 0.8 l. over 3 hours after that. Heating was stopped stirring after concentration and it cooled to ordinary temperature over 2 hours. After-cooling solid-liquid was separated, the after-cooling concentrate was set to C, Cu and nickel were analyzed, and the Cu/nickel ratio was calculated.

[0015]As shown in Table 1, nickel was hardly contained in the precipitate B which heated A and deposited, but high grade copper sulfate (copper sulfate D) with very as little nickel as [ 0.0004 % of the weight ] was obtained by the copper sulfate which made this precipitate remelt and recrystallize, i.e., a real target, by one step of concentration.

[0016]Although the pH of the solution was changed and the equivalent examination was done further, It turned out that it does not generate whether pH at the time of the dissolution remelts this crystal or less by 1.7 in general, and copper precipitates as copper hydroxide, and pH also generates precipitate of nickel hydroxide or more by 4.5 in general, and the refining effects are lost. When it adjusted so that sulfuric acid etc. might be \*\*\*\*(ed) and it might become 1.7 or less about pH even if precipitate generated, it also checked collectively that precipitate remelted.

(Comparative example) What added pure water to 1.3 kg of copper sulfate crystals like the example, and was 2 l. was prepared, and it was considered as the solution A of H<sub>2</sub>O. It stirred slowly, covering a container in a comparative example, so that the

solution A may not consider contamination of air as evaporation, and dissolved thoroughly at about 70 \*\*. No precipitate was generated at this time. It condensed to 0.8 l. over 3 hours after that. Heating was stopped stirring after concentration and it cooled to ordinary temperature over 2 hours. After-cooling solid-liquid was separated, Cu and nickel were analyzed, respectively, and the Cu/nickel ratio was calculated. The precipitating copper sulfate crystal was set to E, and the concentrate was set to F. [0017]the copper sulfate crystal E -- again - the recrystallization copper sulfate G obtained by condensing was analyzed and compared. Furthermore this recrystallization copper sulfate was recrystallized further, and the third-time crystal copper sulfate H was obtained.

[0018]

(表 1)

	単位	Cu	Ni	Cu/Ni
水溶液A	g/l	163	0.256	640
(実施例)				
生成沈殿B	wt%	52.4	0.0014	37430
濃縮後液C	g/l	21.9	0.18	120
再結晶硫酸銅D	wt%	25.5	0.0004	63750
(比較例)				
硫酸銅結晶E	wt%	25.5	0.04	640
濃縮液F	g/l	41.4	0.032	1290
再結晶硫酸銅G	wt%	25.5	0.004	6380
再々結晶硫酸銅H	wt%	25.5	0.0004	63750

As shown in Table 1, nickel is hardly contained in the precipitate B which deposited by A, but making this precipitate remelt and recrystallize, i.e., high grade copper sulfate with very as little nickel as [ 0.0004% ] is substantially obtained by one step of concentration, understands. On the other hand, by the conventional method, three concentration and recrystallization were required to acquire the same nickel grace. [0019]Although it made to obtain a high grade copper sulfate crystal with few nickel contents into the key objective in this invention, as shown in Table 1, it is effective in the copper sulfate solution which nickel condensed after the crystal deposit being obtained. [0020]

[Effect of the Invention]The copper sulfate crystal of a high grade with little nickel comes to be economically obtained by this invention.

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CLAIMS

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[Claim(s)]

[Claim 1]A refining method of copper sulfate collecting copper sulfate which heats in the atmosphere a copper sulfate aqueous solution which dissolved said copper sulfate at not less than 80 \*\*, and carries out separation sedimentation in a refining method of copper sulfate containing nickel, and making said copper sulfate remelt and recrystallize.

[Claim 2]A refining method of the copper sulfate according to claim 1, wherein nickel grace in refined copper sulfate is 0.001 or less % of the weight.

[Claim 3]A refining method of the copper sulfate according to claim 1 or 2, wherein the pH of a copper sulfate aqueous solution before heating is 1.7 or more and 4.5 or less.

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